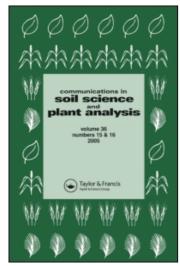
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# Automated Collector of Terrestrial Systems Used for the Gathering of Soil Atmospheric-Gas Emissions

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# **Automated Collector of Terrestrial Systems Used for the Gathering of Soil Atmospheric-Gas Emissions**

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Soil greenhouse gas (GHG) emissions are complex, and their study requires considerable sampling of field spatial and temporal differences. Manual and simple automated gas-collection techniques used at multiple sites during specific time intervals are labor intensive. The objective of this work was to construct a device that can independently collect GHG samples with the accuracy and precision of manually drawn samples. An automated collector of terrestrial systems (ACTS) is a 24-h, 7dlweek programmable sampler used in the field for real-time gathering and containment of soil GHG emissions. The sampler opens and closes an exterior soil gas chamber, mixes gases in the chamber by turning fans onloff, and utilizes programmable circuits to purge the system and draw a sample from the chamber with a pneumatic-driven syringe. Each sample was stored in an evacuated vial held in a 30-vial capacity carousel. Vial content was analyzed for carbon dioxide  $(CO_2)$ , methane  $(CH_4)$ , and nitrous oxide  $(N_2O)$  at the U.S. Department of Agriculture (USDA)-Agricultural Research Services (ARS) Agroecosystem Management Research Unit (AMRU). A Tracor MT-220 gas chromatograph (GC) configured with a thermal conductivity detector (TCD) was used for CO<sub>2</sub> analysis, and an automated gas-sampling system (AGSS) attached to a Varian 3700 GC configured with flame ionization detection (FID) and electron capture detection (ECD) was used for CH<sub>4</sub> and N<sub>2</sub>O analysis. Field and laboratory mean values and coefficients of variation (standards and field concentrations of CO2, CH4, and N2O ranging from ambient to  $71 \text{ kg ha}^{-1} \text{ d}^{-1}$  had coefficients of variation ranging from 1.2 to 4.2%) were similar between ACTS and manually drawn samples. Results showed strong correlation ( $R^2 = 0.81$  to 1.00) between sampling methods. The sampler design provides a realistic and inexpensive approach for collecting emission samples while reducing human error associated with adverse sampling conditions and fatigue. The ACTS has potential for use in monitoring and comparing management practices in terrestrial systems to determine their contribution to GHG emissions.

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Keywords Automated sampler, gas analysis, greenhouse gas emissions, soil, terrestrial systems

#### Introduction

Increasing global population is challenging the agricultural industry to produce sufficient food, fiber, and fuel while conserving soil, water, and air resources. Careful plans to sustain resources include conservation of soil organic matter, minimization of soil erosion, and balancing nutrients for crop production and environmental management (Doran 2005). Global warming concerns and greenhouse gas (GHG) emissions, which contributing to climate change, have prompted world interest. Approximately 20% of global anthropogenic GHG emissions are attributed to agricultural activities. Soils are believed to be one of the major contributors in the agricultural sector. The United Nations Framework Convention on Climate Change (UNFCCC) provides GHG estimates and methods of collection. Measuring soil GHG inventory is relatively more complex than measuring many other GHG sources, and techniques have been implemented only to varying degrees in the UNFCCC (Lokupitiya and Paustian 2006).

The U.S. Department of Agriculture (USDA) conducts and sponsors a broad range of research to improve the understanding of the roles that terrestrial systems play in climate change and the potential effects of global change on agriculture, forest, and range systems with the aim of maintaining and enhancing food, fiber, and forestry production under changing conditions (U.S. Global Change Research Program 2006). It is well known that soil surface carbon dioxide (CO<sub>2</sub>) flux measured at a specific site varies from day to day when sampled at discrete points in time. Automated field chambers used to estimate cumulative CO<sub>2</sub> flux variation indicate that increasing the frequency of sampling from once every 12 days to once every 3 days decreased site-specific variation from +60% and -40% to  $\pm 20\%$  of the expected value (Parkin and Kaspar 2004). When large transient flux events are missed with manual point-in-time sampling, a drastic underestimation of total flux can occur when compared to the sampling at shorter intervals that are possible with automated sampling (Scott, Crichton, and Ball 1999).

The investigation of nitrous oxide (N<sub>2</sub>O) emissions from agricultural soils is confounded by the complex interactions between residue management, tillage, and fertilizer management over varying temporal and spatial systems (Parkin and Kaspar 2006). A recommendation by Six et al. (2004) stated that it is "crucial to further investigate the long-term, as well as the immediate effects of various N-management strategies, such as precision farming, nitrification inhibitors, and type plus method of N fertilizer application, for purposes of long-term reduction of N<sub>2</sub>O-fluxes under notill conditions." The investigation of methane (CH<sub>4</sub>) production and consumption in terrestrial systems (especially agricultural systems) and the effect they have on atmospheric concentrations are not simply characterized. A large number of agricultural systems sampled in Iowa for CH<sub>4</sub> fluxes were considered neutral as a result of observed production and consumption results; however, several fields sampled produced high CH<sub>4</sub> fluxes. Additional sampling indicated municipal landfills in Iowa might contribute significant CH<sub>4</sub> emissions; therefore, it is unclear what effects are from natural or disturbed terrestrial systems. Ongoing studies are providing additional information on specific systems, but clearly more sample collection is needed, especially for agricultural systems (Chan and Parkin 2001).

The complexity of production and consumption of GHG emissions within terrestrial systems and the necessity and difficulty in collecting samples to study global temporal and spatial variability has promoted the development of an automated gas-collection system. Thus, the objectives of this study were to describe the automated collector of terrestrial systems (ACTS) design and compare the precision and accuracy of GHG (CO<sub>2</sub>, N<sub>2</sub>O, and CH<sub>4</sub>) samples collected using ACTS with those samples drawn manually.

# Materials and Methods

#### ACTS Construction

The ACTS (Figure 1) design, used to collect atmospheric air samples for quantitative laboratory analysis, was constructed and tested at the USDA-ARS-AMRU at the University of Nebraska in Lincoln. The device is contained in a watertight Plexiglas box (50 cm long × 50 cm wide × 40 cm high) (Figure 2). A programmable (Microchip Technology Inc. PicBasic Pro Compiler, Chandler, Ariz.) microchip (cat. no. PIC18F458) embedded in the controller board sends a signal to open and close relays, controlling the ACTS components. Motors and pneumatic solenoids are used to regulate compressor airflow (2.1 kg cm<sup>2</sup>) and internal headspace mixing fans (Figure 3). Pneumatic cylinders are attached to different components of the system to perform the tasks necessary to open and close the chamber, actuate a 20-mL sample syringe (LUER-LOK, Becton Dickinson & Co., Franklin Lakes, N.J.; cat. no. 309661) to purge sample lines (Nalgene cat. no. 8000-0002; Thermo Jarrell Ash, Waltham, Mass.), extract a sample from the chamber headspace, pierce vial septa with a needle, inject sample into an evacuated 10-ml vial (cat. no. 223696; Wheaton Industries Inc., Millville, N.J.) sealed with a lyophilization stopper (cat. no. 224100-193; Wheaton Industries Inc.) and an aluminum seal (cat. no. 224178-01; Wheaton Industries Inc.), and advance a 30-vial sample carousel. The components list can be obtained from the corresponding author.

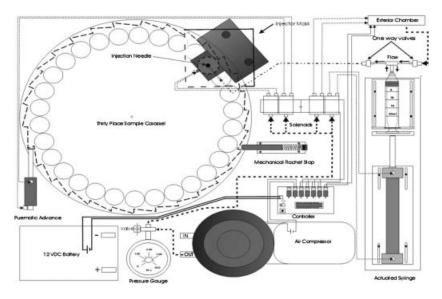


Figure 1. Schematic of ACTS.

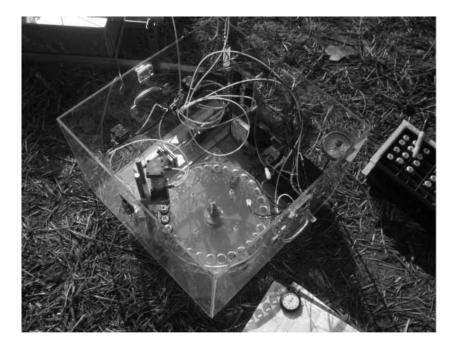


Figure 2. ACTS.

# **ACTS Operation**

Evacuated sealed vials are positioned on a carousel that serves to sequentially position vials under a pneumatic cylinder. The push rod of the pneumatic cylinder is fitted with a side-arm needle fitting connected to a sample line. The sample line is



**Figure 3.** Chamber with internal fans controlled by ACTS.

connected to an exterior chamber. An inline tee on the sample line is separated from the needle and chamber with a check valve. The tee is attached to a pneumatically actuated syringe. At the beginning of a sampling cycle, the exterior chamber is pneumatically closed, fans within the chamber circulate the air, and check valves allow air to be drawn from the chamber and flushed out through the needle. The syringe is activated again, drawing a sample from the chamber. The pneumatic cylinder located over the sample vial is activated, causing the needle to penetrate the vial septum. The syringe is actuated to inject 20 mL chamber gases into the 10-mL vial. The sample needle is actuated, removing the needle from the septum. A pneumatic cylinder advances the carousel to the next vial. After real-time soil flux collection has been completed, the exterior chamber is pneumatically opened, exposing the soil surface to atmospheric conditions until the next sampling cycle.

## ACTS Evaluation

The precision and accuracy of ACTS were evaluated by collecting gas flux from a spring (2 April 2007) soybean stubble field and repetitive collection of certified gas standard (Scotts Specialty Gases), 100% CO<sub>2</sub>, helium (He), and ambient gas. Samples were collected in vials using ACTS, a 30-mL sample syringe to manually withdraw samples (then purged to 20 mL for vial injection), and an automated fourplace spring-powered automatic sampler (AS4) described by Ginting et al. (2007). Collected samples were analyzed for CO<sub>2</sub>, N<sub>2</sub>O, and CH<sub>4</sub> using a gas chromatography (GC; Tracor MT-220) with a TCD and maintained at 110 °C as described by Weier et al. (1993). Nitrous oxide and CH<sub>4</sub> were analyzed by means of an automated gas-sampling system attached to a GC (Varian 3700) as described by Arnold et al. (2001).

A pneumatically actuated chamber (Figure 3) made of aluminum (45 cm long × 39 cm wide × 15 cm high) and designed by T. J. Arkebauer (University of Nebraska at Lincoln) and P. M. Crill (University of New Hampshire) was modified by attaching two fans (New Delta Electronics 12 CFM 12 VDC model EFB0512HA) on opposite interior sides of the chamber. A vent tube (20 cm long × 5.2 mm i.d. vinyl tube) with an optimum diameter and length for selected wind speeds and enclosure volumes as described by Hutchinson and Mosier (1981) was installed. Also, a fourport manifold (www.Smallparts.com; part no. B-TCM-13-20/4-01) attached to four 25-mm Nalgene tubes (cat. no. 14-176-194; Fisher Scientific, Pittsburgh, Penn.) was inserted through a 4.25-mm-diameter hole on top of the chamber. Each of the tubes were attached diagonally to the interior top of the chamber, allowing equal amounts of mixed sample to be drawn from four points inside the chamber. The pneumatic cylinder, fans, and manifold were attached to ACTS to open and close the chamber, mix gases, and collect samples.

Field chambers were placed over rectangular anchors (made from 3.1 mm thick aluminum and measuring 36.5 mm wide, 40.5 mm long, and 10 cm high) that were pressed 7.5 cm deep in the soil. The anchors and chambers were then sealed together with silicone. The chamber used in the laboratory was sealed on the bottom with plastic, and chamber troughs were filled with water to seal gases inside when the lid was closed. A tee was inserted in the sample tubing between the exterior chamber manifold and ACTS one-way valves. The manual sample syringe and AS4 were connected to the tee for comparison of gas-collection methods. The laboratory chamber was flushed with 100% He for 15 min before and after atmospheric air was

introduced. Ten mL of 100% CO<sub>2</sub> was added to the chamber after the second He flush. The ACTS was programmed to run interior chamber fans for 10 s, purge sample lines coming from the chamber with a single 20-mL expulsion, take a sample, and inject the sample into a vial. A sample taken manually using a 20-mL syringe was injected into another vial for comparison of CO<sub>2</sub> concentrations. Samples were drawn on four occasions for each treatment. Carbon dioxide concentrations were increased by 10 mL for each cycle. The preceding sequence was repeated a total of four times. In addition, the chamber was flushed with a certified 602 ppmv CO<sub>2</sub> standard after the last He flush and sample collection.

Six field chambers were placed in a circular configuration between rows. The ACTS was positioned in the center of the circle and attached randomly to each chamber. The sampler was programmed to close the chamber, mix soil gases, and collect samples at 0, 10, 20, and 30 min. In addition, samples were taken manually and with the AS4 each time an ACTS sample was drawn. Samples were injected into 10-mL evacuated vials and analyzed for CO<sub>2</sub>, N<sub>2</sub>O, and CH<sub>4</sub> within 24 h of removal. Sample concentrations were quantified using a GC calibrated with certified standard gases. Flux calculations were made using linear regression of the concentration versus time curve, as reported by Ginting et al. (2003):  $F = k d (273/T) (V/A) (\Delta C/D) (\Delta C/$  $\Delta t$ ), where F is the rate of gas emission (kg ha<sup>-1</sup> d<sup>-1</sup>), k is unit conversion, d is gas density (g cm<sup>-3</sup>) at 273 K and 0.101 MPa pressure, T is the air temperature (K) inside the chamber, V is the chamber volume (cm<sup>3</sup>), A is the area of soil covered (cm<sup>2</sup>), C is gas concentration (percent [v/v] for CO<sub>2</sub>-C and ppm [v/v] for N<sub>2</sub>O-N and CH<sub>4</sub>-C), and t is time interval (minutes between collected samples). The value for k is 1.44  $\times$ 10<sup>6</sup> for CO<sub>2</sub>-C and 144 for N<sub>2</sub>O-N and CH<sub>4</sub>-C to achieve unit cancellation. The values of d are  $5.36 \times 10^{-4} \,\mathrm{g\,cm^{-3}}$  for CO<sub>2</sub>-C and CH<sub>4</sub>-C and  $1.25 \times 10^{-3} \,\mathrm{g\,cm^{-3}}$ for N<sub>2</sub>O-N. Soil cores, temperature, and electrical conductivity (Hanna Inst. conductivity meter, item no. 134489) (Arnold et al. 2005) were sampled or measured between rows at each chamber location. Soil cores taken from each location were analyzed for nitrate (NO<sub>3</sub>) N, ammonium (NH<sub>4</sub>) N, and pH at the AMRU Analytical Laboratory. Statistical analysis of ACTS collected atmospheric gases were compared with manual and AS4 samples using a student t-test with multiple samples (Christian 1986).

## Results

For ACTS to be a useful alternative for in-field gas sample collection, it must meet three requirements. First, mean value concentrations for GHG collected from repetitive sampling must be the same and determined with comparable accuracy when compared to manually drawn samples. Likewise, typical soil flux ranges must yield the same precision and accuracy as manual and AS4 sample collection methods. Second, samples drawn from one cycle to the next cannot contain appreciable differences in residual contamination from the previous sampling. Third, there must be justification for building and operating the sampling device.

In answering the first requirement, ACTS samples collected and analyzed for CO<sub>2</sub> (Table 1) with the exception of the ambient sample, all fell in the 50–80% confidence level when compared to manually drawn samples. When one high pair was removed from the calculation, the ambient sample fell between 80–90%, indicating no significant difference in methods. The high pair of ambient sample that was removed was probably the result of an increase in occupancy of building air used

Table 1. Carbon dioxide concentrations (ppmv) of chamber-collected gas samples using ACTS and using a manual syringe

	$Ambient^b$		100% CO₂ <sup>b</sup>		100% He <sup>c</sup>		602 ppmv CO <sub>2</sub> <sup>d</sup>	
Cycle <sup>a</sup>	Manual	ACTS	Manual	ACTS	Manual	ACTS	Manual	ACTS
1	357.7	349.0	472.7 (1.82) <sup>e</sup>	468.3 (1.33)	5.5	16.2		
2	345.0	342.7	753.7 (2.35)	763.0 (3.50)	15.4	12.6		
3	370.0	362.8	881.3 (2.25)	893.9 (4.17)	20.0	22.4		
4	358.5	358.3	1526.9 (3.60)	1572.1 (5.22)	0.0	12.0		
5	385.4	378.9			2.8	23.7	578.7 (1.2)	574.0 (2.1)
Mean	363.3	358.3	908.7	924.3	8.7	17.4	578.7	574.0
SD	15.19	13.91			8.56	5.44	7.06	11.79
CV (%)	4.2	3.9			98.0	31.3		

 $<sup>^{\</sup>prime\prime}$ ACTS and manually collected chamber gas samples were analyzed for CO<sub>2</sub> concentrations after a 15-min flush with 100% He, laboratory air line, 100% He, and 10 mL of 100% CO<sub>2</sub>. The cycle was repeated four times while adding 10 mL more of 100% CO<sub>2</sub> than the previous cycle. The fifth cycle, a 602 ppmv CO<sub>2</sub> standard (Scotty Specialty Gas, certified value  $\pm$ 5%) was used to flush the chamber in place of the 100% CO<sub>2</sub> added.

<sup>&</sup>lt;sup>b</sup>Mean values of four consecutively collected gas samples for each of four cycles. <sup>c</sup>Mean values of eight collected gas samples for each of five cycles.

<sup>&</sup>lt;sup>d</sup>Mean values of four collected gas samples for cycle 5.

<sup>&</sup>lt;sup>e</sup>Numbers in parentheses are coefficients of variation (%).

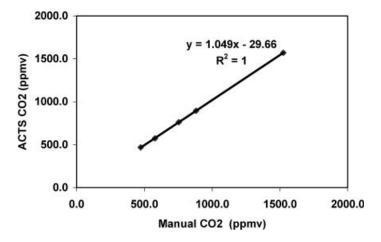


Figure 4. Comparison of ACTS to manual syringe technique for 10-mL increments of 100% CO<sub>2</sub>.

to flush the chamber between samplings. Acceptable coefficients of variation (CV) for  $CO_2$  (1.2–5.2%) resulted when a certified gas standard (Scott Specialty Gases) and 10-mL increases of 100%  $CO_2$  (Table 1) were introduce into the chamber. Likewise, ACTS and manual gas collection of the 10-mL 100%  $CO_2$  increases were correlated (Figure 4). A slope of 1.0 and a linear coefficient of determination ( $R^2 = 1.00$ ) indicated strong agreement between methods. Field soil  $N_2O$  (Figure 5),  $CO_2$ 

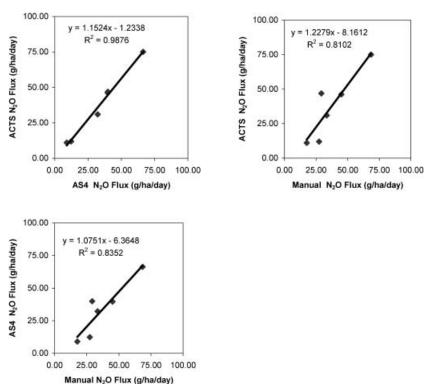


Figure 5. Spring soybean stubble soil flux  $(N_2O)$  comparison of ACTS, AS4, and manual collection of GHG.

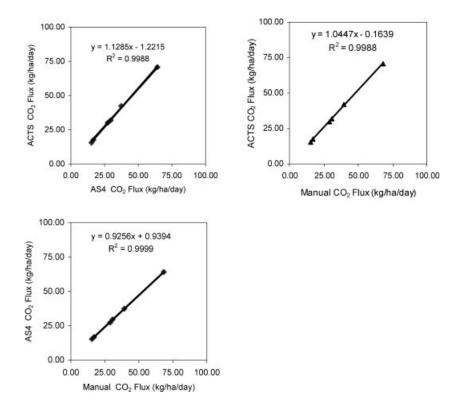


Figure 6. Spring soybean stubble soil flux (CO<sub>2</sub>) comparison of ACTS, AS4, and manual collection of GHG.

(Figure 6), and  $CH_4$  (Table 2) flux comparisons were calculated for ACTS, manual, and AS4 collection methods. Methods correlation for  $N_2O$  (Figure 5) and  $CO_2$  (Figure 6) resulted in slopes of 0.93–1.23 with  $R^2 = 0.81$ –1.00. Methane flux

**Table 2.** Methane concentrations (ppmv) of chamber-collected gas samples in a spring soybean stubble field using ACTS, AS4, and a manual syringe

Chamber <sup>a</sup>	$ACTS^b$	$\mathbf{Manual}^b$	$AS4^b$
1	1.93 (3.75) <sup>c</sup>	1.70 (3.87)	1.94 (2.90)
2	2.12 (1.44)	1.88 (2.37)	1.91 (4.44)
3	2.05 (0.88)	1.76 (5.34)	1.82 (4.59)
4	2.15 (3.22)	1.88 (2.41)	1.87 (4.59)
5	2.04 (1.72)	1.78 (3.47)	1.95 (2.53)
6	2.02 (2.16)	1.83 (4.80)	1.92 (3.68)
Mean	2.06	1.80	1.90
SD	.09	.07	.04
CV (%)	4.1	3.7	2.2

<sup>&</sup>lt;sup>a</sup>Gas samples collected for each chamber at 0, 10, 20 and 30 min using ACTS, AS4, and manual syringe.

<sup>&</sup>lt;sup>b</sup>Mean values for 0, 10, 20, and 30 min.

<sup>&</sup>lt;sup>c</sup>Numbers in parentheses are coefficients of variation (%).

calculation showed neither production nor consumption; therefore no flux was calculated. Instead, mean CH<sub>4</sub> values were calculated (0, 10, 20, and 30 min) for each chamber, resulting in acceptable CV ranging from 2.2 to 4.1% (Table 2). Additional data collected were mean soil (13 °C) and air (25 °C) temperatures, field electrical conductivity (0.49 dS m<sup>-1</sup>), soil NO<sub>3</sub>-N (2 mg kg<sup>-1</sup>), soil NH<sub>4</sub>-N (3 mg kg<sup>-1</sup>), and pH (5.9).

To satisfy the second requirement, 100% He was flushed twice during each of five cycles to remove the varying concentrations of CO<sub>2</sub> that had been added. Helium flushes were sampled a total of eight times during each cycle to measure residual CO<sub>2</sub> (Table 1). Gas chromatography detection limits for CO<sub>2</sub> near zero resulted in a better CV for ACTS (31%) than the manual method (98%). The cycles had CO<sub>2</sub> ranges from 343 to 1572 ppmv, resulting in residual CO<sub>2</sub> ranging from 0 to 24 ppmv. Mean values were used to calculate 1.0% manual and 2.0% ACTS residual CO<sub>2</sub> carryover.

To justify the building and operation of ACTS, one needs to look only at current information, which enables the development of generalizations concerning agricultural and nonagricultural systems and the role they play in GHG production and management. Automated real-time sample collection will allow for a large number of terrestrial systems to be sampled simultaneously without travel time and lengthy waiting at sampling sites. Automated sampling continues during irrigation/rain, temperature, and diurnal cycles. The cost of components to build the ACTS was <\$2,000.

## **Discussion**

Management of agricultural ecosystems to provide sufficient food, fiber, and forestry production while conserving soil, water, and air quality requires additional field investigation to measure spatial and temporal differences. The automated collector of terrestrial systems designed at USDA-ARS-AMRU will facilitate such investigation. The programmable 24-h, 7-d/week soil atmospheric-gas collection device opens and closes an exterior chamber with a pneumatic cylinder and turns electric fans on/off for mixing chamber gases. Chamber gases are injected into evacuated vials, which are removed from the field sampler and analyzed for N<sub>2</sub>O, CO<sub>2</sub>, and CH<sub>4</sub> using a GC equipped with an AGSS. The ACTS watertight design allows for the uninterrupted collection of soil atmospheric gases when fatigue (continuous diurnal/nocturnal sampling) or adverse sampling conditions (irrigation/ rain) exist. ACTS is an inexpensive, portable device that allows undisturbed monitoring of GHG emissions in terrestrial systems. Alternate uses of the ACTS include real-time collection of gases produced in all agricultural (water, crops, crop residue, tillage, fertilizer, lagoons, manure, compost) and nonagricultural (industrial manufacturing, automobile exhaust, etc.) systems.

## **Conclusions**

Increased demands to evaluate agricultural and nonagricultural systems and their contribution to GHG production will facilitate the development of new technology and the improvement of existing technology. Developing a soil atmospheric-gas collection device as described in this article will provide scientists with a precise, accurate, and economical approach to automated gas collection. The systems

program, electronic relays, and pneumatic lines can be easily modified to control additional devices. The ACTS is very versatile and can be used for many different applications.

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